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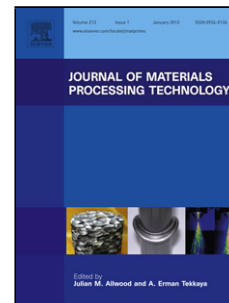
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## **Studies on The Reprocessability of Poly(Ether Ether Ketone) (PEEK)**

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### **Abstract**

Whilst demonstrating desirable mechanical properties, corrosion resistance and the ability to retain structural integrity over extended temperatures, PEEK (Poly (Ether Ether Ketone)) remains expensive, restricting broader usage. The reuse and recyclability characteristics of PEEK are therefore commercially important, where the most prevalent manufacturing process for PEEK is injection moulding. This study comments on the reprocessability of PEEK specifically applied to the injection moulding process, comparing the effect of repeated reuse on mechanical properties. Recycled PEEK retains its tensile properties through at least three moulding and regrinding cycles. XRD and DSC measurements confirmed that reused PEEK shows no degradation in crystallinity.

### **Keywords**

Poly (aryl-ether-ether-ketone); regrind; reprocessing; injection moulding

### **Introduction**

Poly(ether ether ketone) (PEEK) is an engineering thermoplastic polymer known for its high melting temperature, chemical and abrasion resistance, and excellent mechanical properties, particularly toughness (Jones et al., 1985). Consequently, PEEK is used

extensively in high strength and stiffness applications, such as the advanced composite systems reviewed in detail by Nguyen and Ishida (1987). PEEK is now finding ever greater usage in diverse engineering applications including bearings, piston parts, pumps, HPLC columns, compressor plate valves, and cable insulation and ultra-high vacuum systems. It is also extensively used in the aerospace and automotive sectors. Its chemical and wear resistance to hostile environments, along with its ability to withstand thermal sterilisation processes, also makes PEEK a suitable material for medical applications such as orthopaedic and spinal implants (Kurtz and Devine, 2007).

PEEK is however an expensive polymer, currently more than four times the price of other engineering thermoplastics such as PBT, POM and PMMA, so there is a strong economic incentive to recycle PEEK and the industrial recycling possibilities are therefore commercially significant.

To date, recycling studies have focused on PEEK reinforced with carbon fibre (CF). For example, Buggy et al. (1995) used concentrated sulphuric acid to recover PEEK from APC-2 composites, although DSC analysis indicated the presence of impurities in the reclaimed material. For industrial purposes, the favoured approach is mechanical reprocessing. Day et al. (1994) extruded a blend of granulated APC-2 CF/PEEK prepreg offcuts with injection moulding grade PEEK. Test pieces were produced from the blend by injection moulding and were found to have greater tensile strength and Young's modulus than a commercial material containing a similar loading of CF. It was also observed that the viscosity-average molecular weight of the recycled PEEK did not decrease greatly after an additional injection moulding cycle. Sarasua and Pouyet (1997) evaluated the effect of several successive injection moulding cycles on 10% and 30% short carbon fibre (CF) reinforced PEEK.

Damage to fibres as well as degradation of the matrix led to a fall in mechanical properties and the authors suggested that the degradation of PEEK was due to the presence of carbon fibres which may have led to a chemical reaction between PEEK structure and pyrolysis

products of CF. The presence of fillers can therefore hinder understanding of the behaviour of the matrix polymer in recycling operations.

In past studies, the mechanical behaviour of PEEK has been investigated in relation to crystallinity, molecular weight and thermal history. Improvements in Young's modulus and strength together with ductility reductions are generally obtained as crystallinity increases in both neat PEEK and its composites. For example, Chivers and Moore (1994) showed that both modulus and strength of PEEK improve with increasing crystallinity, but the effect was accompanied by embrittlement, as indicated by decreasing toughness. Molecular weight also had an indirect effect, where maximum crystallinity attainable was found to fall at high  $M_w$ . Studies on the effect of thermal history on PEEK crystallisation behaviour (and therefore resulting mechanical properties), have shown that the quality and quantity of the crystalline regions depend on the cooling kinetics and can be altered by the presence of inclusions such as fibres (Sarasua et al., 1996), or by self-nucleation (Jonas and Legras, 1991). Jonas & Legras (1991) also examined the degradation, in terms of branching, induced when held in the molten state, and found that the structural defects created by branching reduced the crystallinity, whilst the restricted molecular mobility associated with increased molecular weight decreased the crystallisation rate. Bakar et al. (1999) reported a 2% drop in the crystallinity of pure PEEK for every 10°C rise in melt processing temperature, although this effect was absent in composites containing 5% or 10% hydroxyapatite. Although Sarasua et al. (1996) observed that pure PEEK maintained its degree of crystallinity during ten successive injection moulding cycles, the mechanical properties of moulded PEEK samples resulting from the successive moulding cycles were not presented in the work, as the main focus of the study was on the short fibre PEEK composites.

In comparison with the lab scale research studies described above which focused on increasing time and temperatures in order to elucidate the degradation mechanisms at work, this study aimed to determine at what point PEEK reuse becomes unfeasible in an injection moulding process, due to degradation and subsequent deterioration in mechanical

performance. To achieve this aim, the effect of reprocessing unfilled PEEK was investigated. Thus, injection moulded PEEK test pieces were reground and processed a further five times by injection moulding to study the effect of successive moulding cycles on the mechanical properties. In addition, first regrind and virgin PEEK were co-processed in varying proportions in order to assess the effect of reprocessed material on the properties of virgin material.

## Materials

The material used for injection moulding was Victrex High Performance PEEK<sup>TM</sup> 450G. All regrind PEEK grades were prepared in-house. Each regrind batch (1<sup>st</sup>, 2<sup>nd</sup>, 3<sup>rd</sup>, 4<sup>th</sup>, 5<sup>th</sup> and 6<sup>th</sup>) was produced by processing the material once in a Battenfeld HM 40/130 injection moulding machine followed by mechanical grinding in order to produce pellets for further reprocessing through injection moulding. The process was repeated six times in order to achieve all six regrind batches. Different percentages of virgin PEEK were then mixed with 1<sup>st</sup> regrind PEEK (virgin/1<sup>st</sup> Regrind) in the following percentages: 100/0; 75/25; 60/40; 50/50; 40/60; 25/75; 0/100 and then injection moulded into test bars.

## Experimental Procedures

### Injection Moulding

The injection moulding parameters used for manufacturing the different percentages of virgin/1<sup>st</sup> regrind PEEK samples and the 1<sup>st</sup>, 2<sup>nd</sup>, 3<sup>rd</sup>, 4<sup>th</sup> and 5<sup>th</sup> regrind batches are presented in Table 1 and Table 2, respectively.

Table 1. Injection moulding parameters used for manufacturing different percentages of Virgin/1st regrind parts

Virgin/1 <sup>st</sup> regrind	Temperatures				Screw Speed (mm/s)	Injection Speed (cm/s)	Injection pressure (bar)	Cooling time (s)
	Die	Zone3	Zone2	Zone 1				
100/0	380	365	365	360	60	100	1210	25
75/25	380	365	365	360	60	100	1210	25
60/40	380	365	365	360	60	100	1210	25
50/50	380	365	365	360	60	100	1210	25
40/60	380	365	365	360	60	100	1210	25
25/75	380	365	365	360	60	100	1210	25
0/100	380	365	365	360	60	100	1210	25

Table 2. Injection moulding parameters applied for manufacturing of 1st to 5th regrind parts.

Material Regrind cycle	Temperatures				Screw Speed (mm/s)	Injection Speed (cm/s)	Injection pressure (bar)	Coolin g time (s)
	Die	Zone3	Zone 2	Zone 1				
Virgin	380	365	365	360	60	100	1210	25
1st	380	365	365	360	60	100	1210	25
2nd	380	365	365	360	60	100	1210	25
3rd	380	365	365	360	60	100	1210	25
4th	380	365	365	360	60	100	1210	25
5th	380	365	365	360	60	100	1210	25

As can be seen in Table 1 and Table 2, no change in the processing parameters was required throughout the moulding work, where all batches could be used to mould good quality parts using the recommended parameters for virgin PEEK moulding.

### **Tensile Testing**

The parts moulded were dog bone shape tensile testing specimens (40 x 5.5 x 2.0mm). Tensile testing was carried out using a LLOYD instruments EZ20 mechanical testing machine. Testing speed for all samples was 10mm/min and gauge length 35mm. 8-10 samples were tested for each batch and the testing was performed at ambient temperature (20°C).

### **X-Ray Diffraction (XRD)**

X-ray diffraction analysis (XRD) was performed on the injection moulded parts directly using a Bruker D8 Advance X-Ray Diffractometer with a LynxEye detector, operating at 40kV voltage and 40mA current using  $\text{CuK}\alpha$  radiation ( $\lambda=0.1542\text{nm}$ ) in the  $2\theta=5^\circ\text{-}35^\circ$  range in  $0.03^\circ$  increments. The specimens had no additional treatment prior to analysis.

### **Differential Scanning Calorimetry (DSC)**

Thermal analysis measurements were carried out using a Mettler-Toledo DSC 821e under a  $60\text{ml min}^{-1}$  nitrogen flow. Both sets of samples (regrind cycles and mixtures of virgin/<sup>1st</sup> regrind) were heated and cooled between  $30^\circ\text{C}$  and  $400^\circ\text{C}$  at  $10^\circ\text{C min}^{-1}$ . Three repeat measurements were carried out on samples cut from the tensile testing specimens. The sample was always taken from the middle and core of the dog bone specimens, in order to avoid producing any additional variation in data due to differences in skin and core

crystalline structure. It is well known that the skin of a moulded sample experiences a more rapid cooling rate after injection moulding which reduces the degree of crystallinity, compared to the slower cooling core, which often demonstrates a higher degree of crystallinity: Shen et al. (2004) reported crystallinity varying by up to 20% between core and surface in the case of PA66/clay injection moulded dog-bone samples due to this effect.

The degree of crystallinity was calculated using the sample melting enthalpies and the melting enthalpy of 100% crystalline PEEK sample, taken as 130J/g as published in the literature (Blundell and Osborn, 1983).

## Results and Discussion

### Mechanical Properties

Figure 1 shows the tensile strength and elongation at break of mixtures of virgin and 1<sup>st</sup> regrind of PEEK. Tensile strength, as shown in Figure 1a, was independent of the blend composition and therefore was not significantly affected by the combination of virgin and regrind material. More variation was seen in the elongation at break (Figure 1b); although the higher variation within samples, as shown by the error bars, indicated that there was no significant difference between treatments. It is therefore evident that no significant changes in the tensile properties were encountered across the range of samples; from 100% virgin PEEK to 100% regrind PEEK.

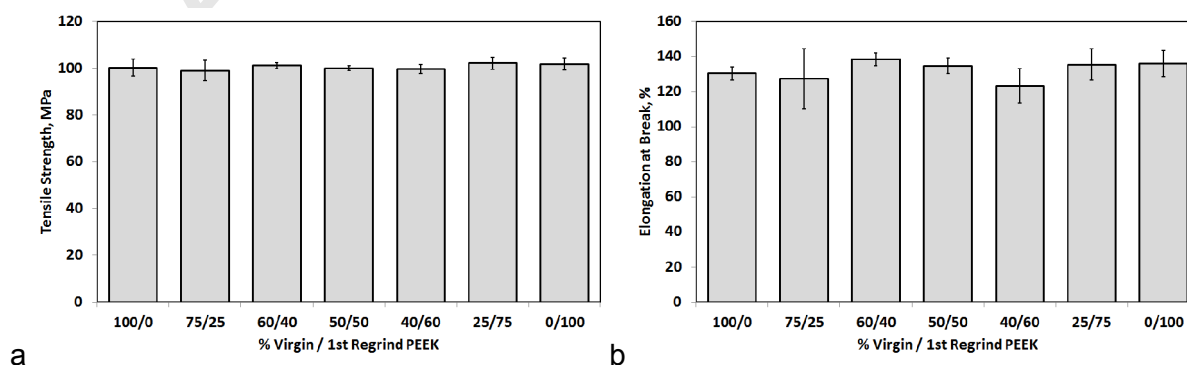




Figure 1. (a) Tensile Strength and (b) Elongation at Break of Virgin/1st Re grind % PEEK (100/0; 75/25; 60/40; 50/50; 40/60; 25/75; 0/100).

The variation in tensile strength and elongation at break of PEEK over multiple processing (moulding/regrinding/re-moulding) operations is presented in Figure 2. The tensile strengths of first and second regrind PEEK specimens (Figure 2a) were not significantly different from that of virgin PEEK, whereas the tensile strengths of third, fourth and fifth regrind PEEK samples were about 10% lower than virgin PEEK. PEEK reprocessed over multiple cycles showed significant differences in values for elongation at break. The elongation at break decreased by 25% after the 3<sup>rd</sup> cycle and 40% by the 4<sup>th</sup> cycle, compared to virgin PEEK (Figure 2b). This experiment demonstrated that in a standard injection moulding manufacturing process, where the material passes through the injection moulding barrel in approximately 20 minutes at a temperature below 400°C, first regrind PEEK does not suffer any major degradation changes and therefore 100% reuse is possible. Thus neither of the two degradation parameters, cross-linking or chain scission, are significant enough to influence the mechanical performance.

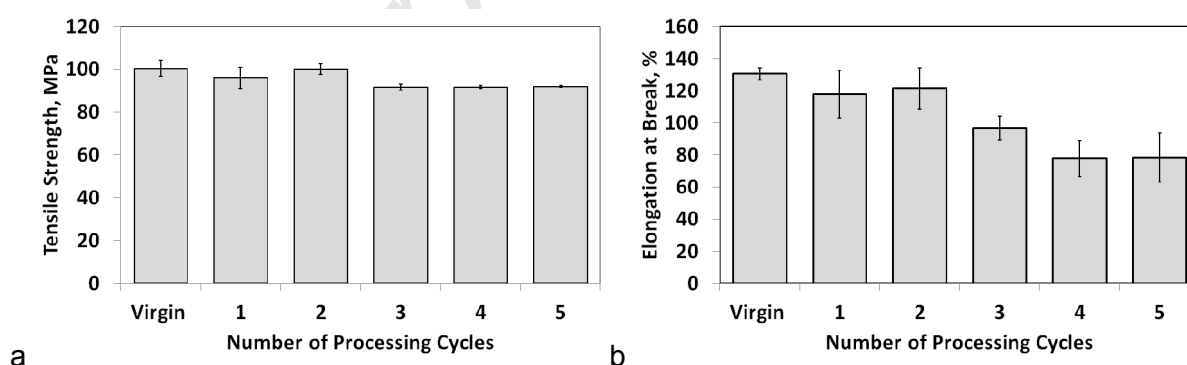


Figure 2. (a) Tensile Strength and (b) Elongation at Break of Re grind PEEK after 1st; 2nd, 3rd, 4th, 5th processing cycles.

Oxidative degradation of PEEK includes two reactions, chain scission and cross-linking, where chain scission leads to shorter molecular chains with higher mobility, which allows

rearrangement of the crystalline structure, whereas cross-linking leads to molecular branching and netting. Investigation of the crystallisation on a hot stage microscope (Jones et al., 1985) by prolonged heating of PEEK above 673K showed that both nucleation and growth of spherulites is reduced, which implies reduced crystallinity, in favour of the formation of a cross-linked structure. Similarly Day et al. (1990) showed that PEEK processing at longer times and higher temperatures in air, leads to a lower level of re-crystallisation material and concluded that the oxidative reactions appear to be the precursor to cross-linking reactions. The spectroscopic evidence suggested that the chain cleavage occurs adjacent to the carbonyl functional groups and the cross-linking takes place between adjacent aryl rings. Patel et al. (2010) carried out a detailed review of PEEKs decomposition mechanisms and products, by inference through analysis of volatile organic products at differing temperatures. It was stated that PEEK has superior thermal degradation resistance, with a continuous use temperature of 260°C and a melting point of 343°C, where the onset of thermal degradation resulting in mass loss only starts between 575-580°C. Between 343 -575°C, low mass loss, cross linking and chain scission dominate. This latter temperature range encompasses the processing temperature range used in our study, therefore we conclude that the embrittlement of the PEEK after the third processing cycle can reasonably be explained by the effect of cross-linking reached through reheating of the material becoming less dominant and the effects of chain scission becoming more significant. Furthermore, the processing temperature was not high enough to bring about mass loss resulting from thermal degradation.

### **Thermal Behaviour and Crystallinity**

The effect of incorporation of different percentages of 1st regrind PEEK in virgin PEEK was investigated by following the crystallisation exotherm values (see Figure 3a). Similarly, the effect of number of reprocessing cycles on the crystallisation exotherm values was also

calculated, as is presented in Figure 3b. The enthalpies of crystallisation in both graphs show no significant change indicating that the amount of crystalline material remains the same independent of the percentages of virgin/1<sup>st</sup> regrind mixes and number of processing cycles. Jonas and Legras (1991) showed that PEEK maintained at 385°C still retained numerous crystallisation nuclei; therefore the samples in our experiment may have maintained a high population of nucleants. If the degree of crystallisation remains fairly constant, it would be expected that tensile properties should also remain stable, as indicated in past studies. Chivers & Moore (1994) showed a linear correlation between tensile yield stress and degree of crystallinity in PEEK films, while Sarasua and Pouyet (1997), looking at PEEK composites, found similar trends in both neat PEEK and its composites, where Young's modulus and strength improve as degree of crystallinity increases. This behaviour is borne out in the mechanical data.

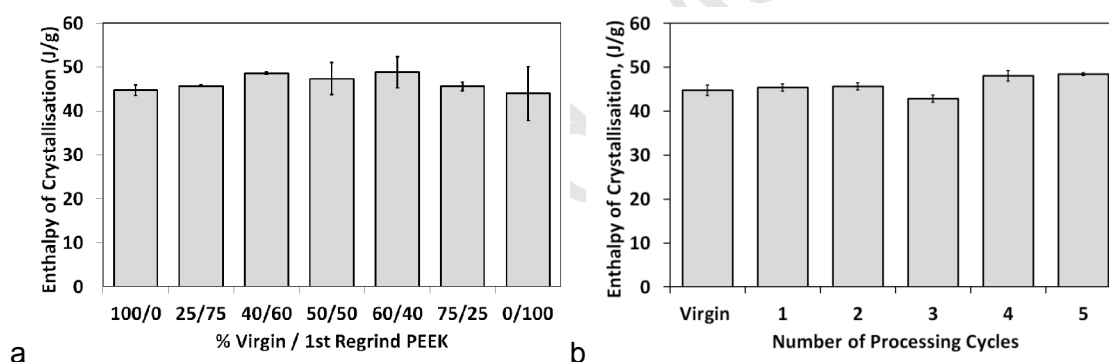


Figure 3. Enthalpy of crystallisation (J/g) of PEEK of (a) Virgin/1st Regrind mixes and (b) virgin, 1st; 2nd, 3rd, 4th, 5th processing cycles.

Similarly, the crystallisation temperatures measured for both sets of data (in Figure 4a and b) show no significant changes. This is consistent with the observations of Jonas and Legras (1991), who concluded that the crystallisation onset temperature was independent of an isothermal treatment time in the temperature range 400°C to 420°C. It is therefore reasonable to conclude that a hold time of 20 minutes at 385°C, as used in this experiment,

would similarly show no effect on crystallisation onset. It has also been shown by Jonas and Legras (1991) and Day et al. (1990) that an increase in branching and cross-linking mechanisms during degradation led to a reduced molecular mobility and lower crystallisation temperatures. These studies also reported significant decrease in enthalpies and temperature of crystallisation when PEEK samples were held at temperatures above the melting temperature. However, Day et al. (1990) observed that there is also an induction time before which no decrease in crystallisation enthalpy takes place and it was concluded that this does not necessarily mean that no degradation takes place, but that there were insufficient defects along the polymer chain to cause a decrease in crystallinity. Similarly, Hay and Kemmish (1987) reported a reduction in crystallinity brought about by cross-linking of PEEK only when heated above 400°C.

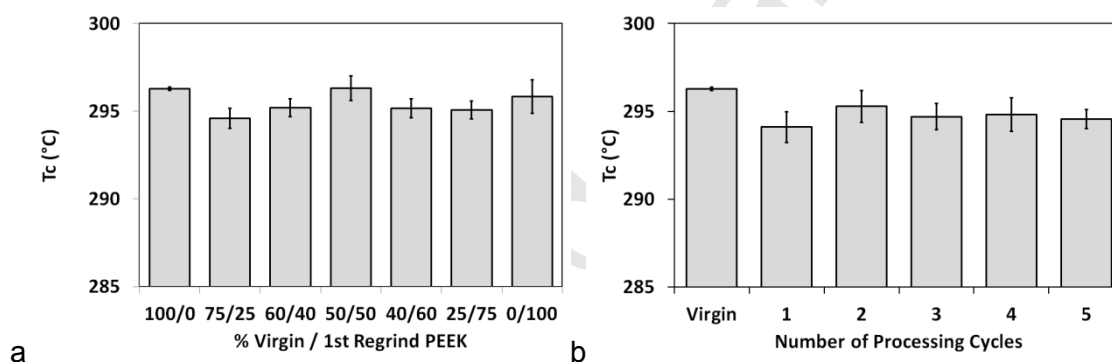


Figure 4. Crystallisation temperature ( $T_c$ ) of PEEK samples (a) Virgin/1st Regrind mixes and (b) virgin, 1st; 2nd, 3rd, 4th, 5th processing cycles.

From these observations it can be concluded that simply cycling the PEEK material through its melting temperature during the manufacturing process, even repeatedly, is not sufficient to produce significant thermal degradation and affect mechanical performance. Rather, it is isothermal conditioning of PEEK over prolonged periods which seems to lead to strong degradation behaviour. However, this effect is not significant in the injection moulding processes reported herein.

### Evaluation of Crystallinity (DSC, XRD)

Table 3 and Table 4 present the degree of crystallinity ( $X_c$ ) of virgin/1<sup>st</sup> regrind PEEK mixtures and the crystallinity of 1<sup>st</sup> to 5<sup>th</sup> regrind PEEK processing cycles, respectively, as calculated by DSC and XRD. All specimens were analysed by DSC, but XRD analysis was confined to three specimens selected from each treatment regime and was performed in order to confirm that the trends observed within the DSC data represented a real effect.

Table 3. Percentage crystallinity (%  $X_c$ ) as calculated by DSC and XRD for virgin/1st regrind samples.

% Virgin/1 <sup>st</sup> regrind	$X_c$ % (DSC)	$X_c$ % (XRD)
100/0	31.3 ± 1.7	24.6 ± 1.4
75/25	30.7 ± 1.7	
60/40	32.7 ± 0.6	
50/50	36.3 ± 4.1	23.9 ± 1.04
40/60	29.5 ± 0.5	
25/75	31.2 ± 0.8	
0/100	30.0 ± 2.8	24.2 ± 1.4

Table 4. Percentage crystallinity (%  $X_c$ ) as calculated by DSC and XRD of 1st to 5th processing cycles.

No of processing cycles	$X_c$ % (DSC)	$X_c$ % (XRD)
Virgin PEEK	31.3 ± 1.7	24.6 ± 1.4
1 <sup>st</sup>	33.1 ± 1.7	24.7 ± 1.6

2 <sup>nd</sup>	27.5 ± 1.7	
3 <sup>rd</sup>	30.6 ± 0.3	25.7 ± 1.8
4 <sup>th</sup>	31.1 ± 2.0	
5 <sup>th</sup>	28.9 ± 1.9	24.6 ± 1.0

As can be seen in Table 3 and Table 4, the DSC and XRD crystallinity data of both batches of PEEK show very little change when compared with virgin PEEK. If crystallinity does indeed make a major contribution to mechanical properties, then this is consistent with the maintenance of tensile strength across the processing cycles. However, the crystallinities obtained by X-ray diffraction are somewhat lower than those obtained by DSC measurements. This is not surprising as the XRD measurements are measuring the crystalline fraction based on the separation of oriented crystalline and un-oriented non-crystalline (i.e. amorphous) scatter, through a curve fitting procedure which gives significant room for error. In addition, XRD can be considered to be a 'static' measurement in that the crystalline structure of the specimen does not change during the course of the measurement, whereas during DSC analysis, the specimen is heated through its glass transition temperature and additional transitions such as cold crystallisation during heating cannot be ruled out entirely (Buggy and Carew, 1994). Other authors such as Hsiao et al. (1993) and Shen et al. (2004) have also reported differences in crystallinity values calculated using the two methods and attributed the discrepancy to changes in crystallinity occurring during the DSC scan. Despite this potential source of error, Zimmermann and Könnecke (1991) concluded that DSC was the best method by which to determine crystallinity in PEEK and the related materials, poly(ether ketone) (PEK) and poly(ether ether ketone) (PEEK). Indeed it could be argued that observation of the dynamic changes during DSC yields further information about the nature of the material which would not be obtained by XRD analysis alone.

The results obtained in Table 3 and Table 4 were combined in Figure 5 in order to investigate further whether there is any correlation between the crystallinity values obtained from XRD and DSC and the gradual change in the PEEK material (either through incorporation of different percentage of 1<sup>st</sup> regrind or repeat reprocessing). The results in Figure 5 appear clustered together with no apparent linear correlation, as other research studies have reported (Karacan, 2005). This clustering effect confirms once again, that there are no significant changes at molecular level due to incorporation of percentages of 1<sup>st</sup> regrind or repeat reprocessing.

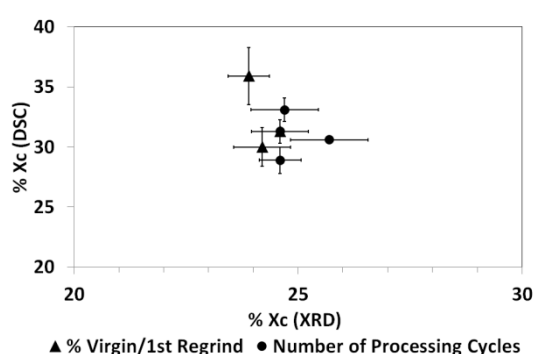


Figure 5. Correlation between crystallinity values obtained using DSC and XRD.

## Conclusions

It has been demonstrated that it is possible to reprocess PEEK within an injection moulding process, whereby it has been shown that first regrind injection-moulded PEEK demonstrated similar mechanical properties to virgin PEEK. It has also been shown that at least three moulding/regrinding/re-moulding cycles can be performed without an appreciable drop in tensile strength, although after the third cycle there was some reduction in elongation at break. This reduction was due to the deleterious effects of chain scission becoming dominant over the strengthening effect of cross-linking occurring at the processing

temperatures used in this experiment. DSC was used to investigate changes in crystallisation temperature or degree of crystallinity, with no appreciable differences found in any of the batches examined. The maintenance of crystallinity explained the retention of tensile strength through at least five regrind cycles. These findings were confirmed by selected XRD measurements, although a direct correlation between the two techniques is difficult to establish because of the changes that can occur during DSC analysis. However we concur with Zimmermann and Könnecke (1991) that DSC is the most useful method for studying the crystallinity of PEEK and related materials.

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## Highlights

- PEEK was reprocessed five times by regrinding and injection moulding
- First regrind and virgin PEEK were blended in varying proportions
- The tensile properties of PEEK were maintained through three regrind cycles
- Regrind PEEK had no significant effect on the tensile properties of virgin PEEK
- Regrinding and blending had no significant effect on PEEK crystallinity